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THE PRODUCTION OF AN ASPHALT RESEMBLING GILSONITE BY THE DISTILLATION OF A MIX- TURE OF FISH AND WOOD.

(Plate X.)

BY WM. C. DAY.

(*Read May 20, 1898.*)

A few years since, I undertook a rather detailed experimental study of the variety of asphalt known as gilsonite,¹ which is mined for commercial use in Utah. Gilsonite is a black, glistening, brittle material, yielding a dark-brown powder when finely pulverized. It fuses readily, becoming a liquid which begins to boil at a temperature above the limit of a mercury thermometer.

It is entirely soluble in carbon bisulphide, not entirely soluble in ordinary ether, partly soluble in absolute alcohol, petroleum ether, glacial acetic acid and chloroform, imparting to these solvents a yellowish to red color with green fluorescence. Besides carbon and hydrogen, it contains sulphur, nitrogen, a trace of oxygen and one-tenth of one per cent. of ash.

Among the various products which I obtained by distilling gilsonite may be mentioned as of interest in this connection certain nitrogenous bases extracted from the distillates by the action of dilute acid and precipitated therefrom by alkalies. These bodies have an odor like that of the pyridine and quinoline series. Such substances were first obtained from bitumen by Prof. S. F. Peckham, who noticed them in distillates from California petroleum; later by myself from an asphalt occurring in Coos county, Oreg., also in the product which forms the subject of this paper.

As a result of considerable experimental work in the past few years with asphalts from a variety of sources in the United States, together with a study of the literature pertaining to the question of the origin of the bitumens from both the geological and the chemical standpoints, I became impressed with the belief that the solid and also some of the higher boiling liquid bitumens have been formed in the earth by the distillation of mixed animal and vegetable material, together with steam at high temperatures, but at pressures which may or may not have been high. Petroleum distillates have been obtained by Warren and later by Engler from fish oil,

¹*Journal Franklin Institute*, Vol. clx, p. 221.

and still more recently by Sadtler from linseed oil. In addition to liquid distillates, paraffin has also been obtained by these investigators. No mention, however, of an analogous production of asphalts, so far as I am aware, has ever been made.

To test the correctness of the belief already expressed, I tried the following experiments:

Into a cylindrical iron retort were introduced a number of fresh herring, a quantity of pine saw dust and a number of small pieces of fat pine wood. The retort was connected by plaster-of-Paris joints with a short glass tube, and this with a gas pipe four feet long, the latter being placed in an ordinary combustion furnace, the other end of the pipe was connected with a Liebig's-cold-water condenser.¹ After charging and closing the retort, it was heated by means of gas stoves, which together with the retort were surrounded with loose bricks to prevent the loss of heat. The heating of the retort was regulated by the rapidity with which vapors were evolved, an increase of heat being necessary toward the end of the distillation. The gas pipe was simultaneously heated to bright redness by the combustion furnace. The pressure was that of the atmosphere. During the progress of the distillation water and oil together with a white smoke flowed from the condenser into the receiver. The oil obtained was lighter than water, of bad odor and very dark red in color. At the end of the gas pipe next to the retort carbon separated, and on one occasion nearly choked the pipe. Only once was an oil heavier than water obtained, and this was small in amount.

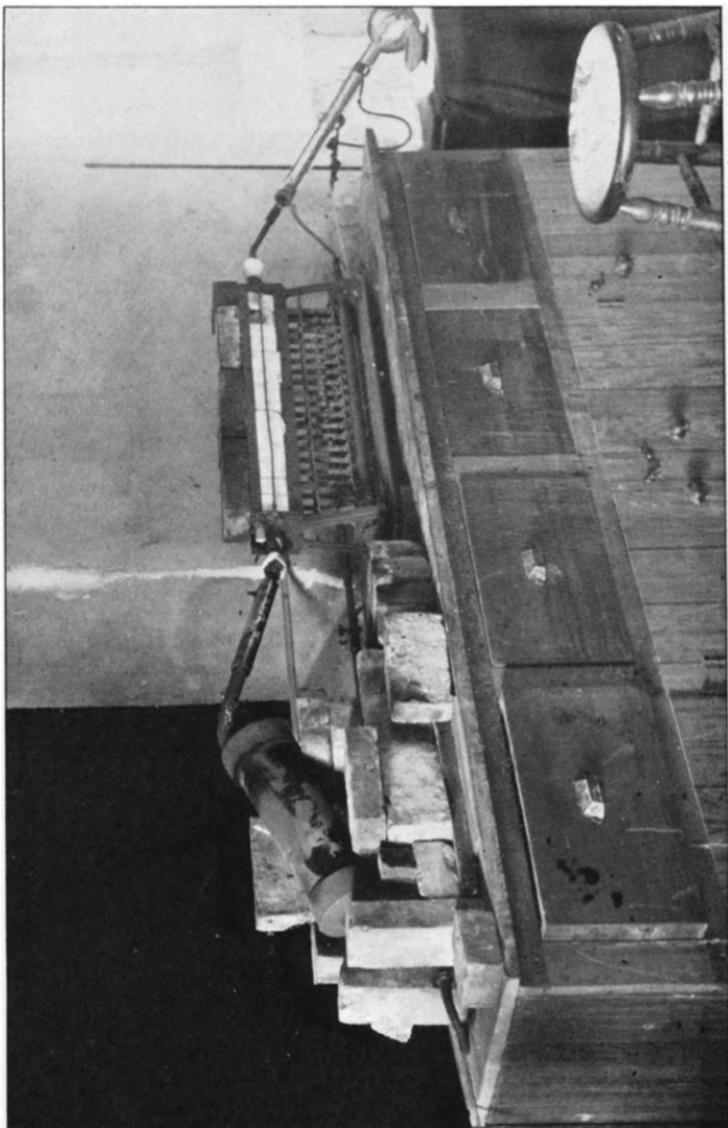
The condensed oil was separated from the water on which it floated, and finally completely dried over chloride of calcium.

It was then placed in a distilling bulb provided with thermometer and distilled, using a straight glass tube as an air condenser.

Boiling began at about 100 Centigrade, but the mercury soon rose to 120. The distillate between these limits consisted of a lemon-yellow mobile oil together with a few drops of water.

At 120 C. the receiver was changed, and another fraction darker in color and less mobile was obtained while the mercury rose to 180. The third fraction was collected between the limits 180 and 245; the fourth between 245 and 315, this fraction showing a pronounced greenish fluorescence, the color by transmitted light being

¹ See accompanying photograph.



dark red. At 340 the receiver was again changed, the thermometer removed and the distillation continued until the temperature was (at a guess) about 425 Centigrade. At this point the distilling bulb cracked and the operation was stopped. On cooling, the contents of the bulb became a black brittle solid, showing a very pronounced resemblance to gilsonite in every way. The following are its properties : Black glistening color, becoming brown on pulverizing, and slightly darker than gilsonite ; fracture conchoidal, entirely soluble in carbon bisulphide ; ether dissolves 90.6 per cent. ; alcohol 66.3 per cent. ; petroleum ether 61.1 per cent. All these solutions show greenish fluorescence, while the color by transmitted light varies from yellow to reddish.

As already stated, the distilling bulb cracked before I had decided to stop the distillation, and the solid product being slightly sticky to the touch, I distilled a second portion of oil, collecting the same fractions as before, but continuing the heating longer. This time I obtained a solid so like gilsonite that it was difficult to tell which was which. A combustion of the first sample gave carbon, 87.5 per cent., and hydrogen, 7.7 per cent. A combustion of the second sample gave carbon, 88.9 per cent., and hydrogen, 6.7 per cent. The figures for Utah gilsonite are 88.3 for carbon and 9.9 for hydrogen. At the time (three years since) I made the analysis of gilsonite nitrogen was not determined. Recently I have determined the nitrogen, and found it to be 1.96 per cent. Since the carbon, hydrogen and sulphur figures added up to 99.5 one or all of the three must have been high, and I am inclined to think that it was the hydrogen, since I cannot now be sure that the gilsonite sample was entirely dry.

The agreement in properties and composition between the gilsonite and my product is much more perfect than it would have been reasonable to expect at the outset of the experiment, particularly when it is remembered that both are doubtless mixtures in themselves, and that certainly on distillation they yield highly complicated mixtures of hydrocarbons.

As the product under discussion was obtained only a few days ago, I have not had time to carry the investigation further, but enough has been done to show how a solid asphaltum may have been formed in nature and to afford a rather satisfactory demonstration of the correctness of views entertained by a number of scientists who have formed their opinions largely on geological evidence and the study of natural occurrences.

It should be stated, in addition, that for the sake of comparison, fish alone, *i. e.*, without wood, were subjected to distillation and without passing the vapors through the red-hot tube. The products obtained included nothing that bore any resemblance to gilsonite and they were radically different from the products which accompanied the distillation of fish and wood together.

This brief paper is of course only preliminary to one which should consider the literature of the subject more in detail, and which may, I trust, throw some light on a few of the many problems which naturally suggest themselves.

I shall continue the work as rapidly as my limited time and facilities for such study will permit. In conclusion I take pleasure in expressing my obligations to my assistant, Mr. Eugene Leamy, for his very intelligent and effective aid in carrying on the experimental work.

SWARTHMORE COLLEGE, May 19, 1898.